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Pinning of elastic ferromagnetic/antiferromagnetic interfaces in phase-separated manganites

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Abstract
We present a study of the magnetic properties of the $Pr_{0.5}Sr_{0.5-x}Ca_xMnO_3$ manganite ($x = 0.1$ and 0.2) in the temperature region where phase separation occurs. This state is characterized by the presence of ferromagnetic (FM) inclusions inside an antiferromagnetic (AFM) matrix. The evolution of the magnetization (M) with magnetic field shows the existence of a critical field, H_C, above which M rapidly increases, indicating a sudden expansion of the FM volume against the AFM one. We analyze this behavior and the response of the magnetic susceptibility at low fields ($H < H_C$) in terms of a thermally activated motion of pinned FM/AFM elastic interfaces. The pinning mechanism is likely to be related to the martensitic accommodation strain around the magnetic and structural interfaces. From this analysis we estimate the size of the FM domains and the parameters that characterize the pinning potential.

1. Introduction

The observation of the colossal magnetoresistance (CMR) phenomenon in the manganese perovskites has promoted a great deal of activity in the area of magnetic oxides in recent decades [1–6]. Accumulated evidence has led to the proposal of phase separation (PS) as the underlying mechanism governing the CMR behavior in mixed-valence manganites [6–9]. This has triggered important research work focused on this topic, especially on the coexistence between ferromagnetic metallic (FM) and charge-ordered antiferromagnetic (CO-AFM) phases. Early in the 1950s, Wollan and Koehler suggested the existence of PS in the $La_{1-x}Ca_xMnO_3$ compound [10]. Nowadays, the accessibility of new experimental techniques has allowed a direct observation of the phase coexistence, namely by scanning tunneling microscopy [7], electron microscopy [8, 9, 11], NMR [12, 13], neutron scattering [14–18], or even by magnetic and transport measurements [19–22].

In spite of this investigation boost toward the understanding of CMR manganites, a conclusive scenario for the occurrence of PS is still lacking. Some authors proposed that a spontaneous spatial segregation of the charge carriers could be responsible for the observed PS [6, 23–25]. It seems feasible that this type of electron-driven PS occurs in lightly doped compounds, where polaronic-like PS was observed at the nanometer scale [18]. For heavily doped materials, on the contrary, the observation of clusters of hundreds of nanometers makes the charge-segregation-type PS unlikely [7, 8, 11]. On the other hand, it has been shown that random disorder can account for the PS at the sub-micrometer scale [21, 26, 27]. One of the proposed candidates as a disorder promoter is the A-site mismatch, σ_A^2 [6, 27]. The different ionic sizes of the cations occupying the A-site of the perovskite structure induce local distortions that affect the energy balance between the competing phases, thus opening the possibility of phase coexistence [28, 29]. Finally, we must mention that the FM and CO-AFM phases show important differences between their lattice structures [14]. As a result, considerable lattice strain and structural distortions appear in the neighborhood of the FM/AFM interfaces (the domain walls separating the FM and CO-AFM phases). Therefore, the FM $\leftrightarrow$ AFM phase transitions could present martensitic characteristics, since the motion of the magnetic interfaces would also imply small atomic displacements, i.e. the domain walls must also drag their associated structural distortions. This would lead to the self-trapping of the

FM/AFM interfaces by the energy barriers imposed by their own strain fields, thus freezing the domain wall dynamics and allowing the presence of both phases. Indeed, this kind of martensitic effect has already been found in some numerical calculations [30], low-temperature magnetization and electron microscopy experiments [11, 31], as well as with other related experimental techniques [32].

In phase-separated manganites, the application of a magnetic field (H) induces the swelling of the FM clusters due to the gain of the Zeeman energy [7, 21]. Consequently, the percolative electronic transport is favored and the resistivity is strongly reduced, producing the CMR effect [5–8, 33–35]. In this context, the pinning of the magnetic interfaces could play a relevant role in the dynamics of the FM domains, thus the study of the evolution of the FM domains under the application of an external magnetic field is of utmost importance.

In this work, we focus our attention on the magnetic properties of the $Pr_{0.5}Sr_{0.5-x}Ca_xMnO_3$ compound for $x = 0.1$ and 0.2. For temperatures below ∼200 K these samples present the coexistence of the FM and CO-AFM phases. In this temperature (T) range, the magnetization versus field loops exhibit two different field regimes. At low fields we observe a thermally activated response with a slow dynamics of the FM clusters, while for H above a critical field H_C a rapid and irreversible increase of the FM volume occurs. We discuss this behavior in terms of the pinning of elastic FM/AFM interfaces, and its possible relation to the strain-induced self-trapping and quenched random disorder.

2. Magnetization results

The $Pr_{0.5}Sr_{0.5-x}Ca_xMnO_3$ polycrystalline samples ($x = 0.1$ and 0.2), were prepared by the nitrate decomposition route, as reported elsewhere [28]. Neither x-ray diffraction (XRD) nor neutron powder diffraction (NPD) experiments show any impurities in these samples. However, NPD in the sample $x = 0.2$ has revealed the presence of multiple crystallographic perovskite phases, each of them clearly associated with the different magnetic phases present in this material [14].

For the present study, magnetization (M) data were measured in a Quantum Design Squid magnetometer equipped with a 50 kOe magnet. $M(H)$ curves were obtained on warming after cooling the samples in zero field from 300 to 50 K. The M versus H results for the sample $x = 0.1$ at three representative temperatures are shown in figure 1 (results for $x = 0.2$ are qualitatively similar, [21]). These temperatures (50, 150, and 180 K) correspond to the region where the PS between the FM and CO-AFM phases occurs [21], as confirmed by neutron scattering experiments (NPD also shows a tiny amount of the A-type AFM phase) [14]. In figure 1 we observe, close to zero field, the typical response of FM systems, with a steep increase of magnetization from the virgin condition corresponding to the initial spin alignment within the FM volume. However, once the system is magnetized a linear behavior with small slopes is obtained. The straight dotted lines are fits to this linear regime. From the slope of these lines we obtained the susceptibility (χ),

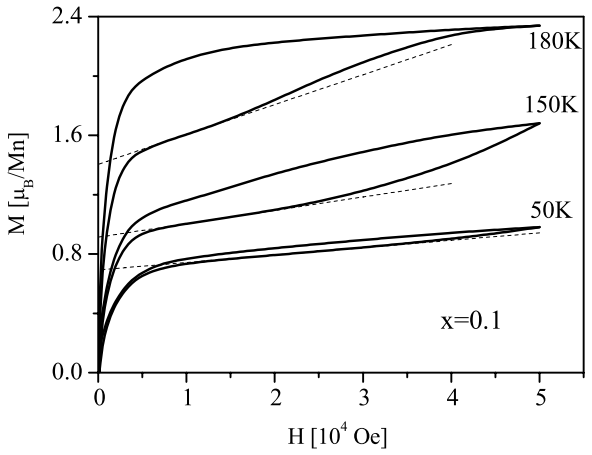

Figure 1. M versus H curves at different temperatures for the $x = 0.1$ sample (a complete set of $M(H)$ data for both samples can be found in [21]). The dotted lines show the linear regime of the magnetization.

and from the back-extrapolation to $H = 0$ the spontaneous magnetization M_0, which is proportional to the zero-field FM phase fraction (∼20% at low T, [14, 21]). In these loops, when the applied field exceeds a critical field H_C, the magnetization splits up from the linear behavior, exhibiting a large hysteresis when H decreases again. This behavior is similar to that observed in other phase-separated manganites, where sharp metamagnetic transitions with martensitic characteristics occur at very low temperatures [31].

To clearly illustrate this behavior, in figure 2 we show the difference ΔM between the measured magnetization and that calculated from the linear fits in figure 1. The rapid departure of M from linearity is now evidenced by the increase of ΔM for $H > H_C$. From these curves we estimate the critical field at each temperature, as exemplified in figure 2(b) for the sample $x = 0.2$ at 170 K. The temperature-dependent critical field, $H_C(T)$, is shown in figure 3 for both samples along with a T–H phase diagram previously obtained from magnetic and transport measurements [21], where the PS region is indicated.

3. Pinning of elastic interfaces: model and discussion

The above-described results strongly suggest that H_C is the field necessary to overcome some pinning forces that oppose the swelling of the FM clusters. We assume that the domain walls (DWs) separating the FM domains from the AFM background are somehow trapped; thus when the field is high enough the DWs are pulled out from the pinning potential and an abrupt increase of the FM clusters occurs, producing the sudden increase of magnetization.

For $H < H_C$, the linear $M(H)$ curves should be related to a very slow dynamics of the DWs in a disordered environment, where a small fraction of the magnetic and structural interfaces would be able to escape from the pinning sites assisted by the thermal energy. Indeed, this picture is supported by the temperature dependence of the low-field susceptibility, obtained from the linear fits of figure 1. In

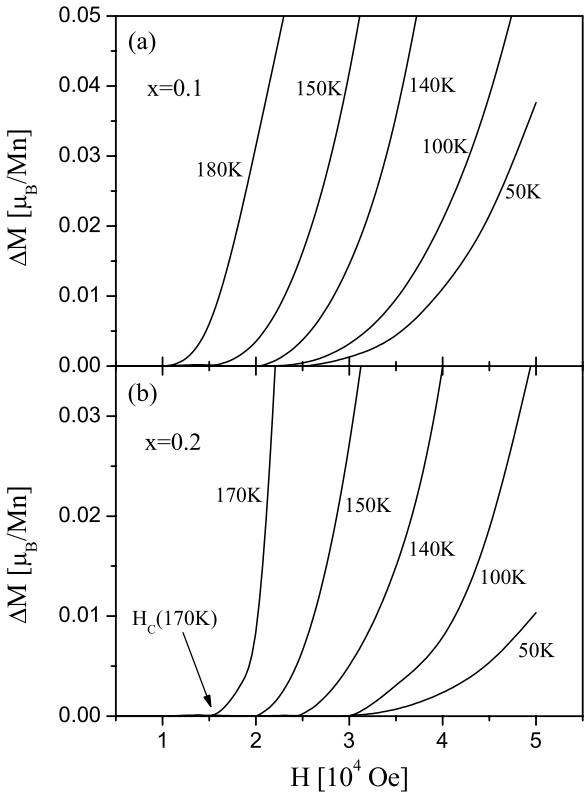

Figure 2. Magnetic field dependence of the difference ΔM between the measured magnetization and the linear fits obtained below the critical field H_C (see figure 1). (a) Sample $x = 0.1$ and (b) $x = 0.2$. Each curve corresponds to a different temperature, as labeled.

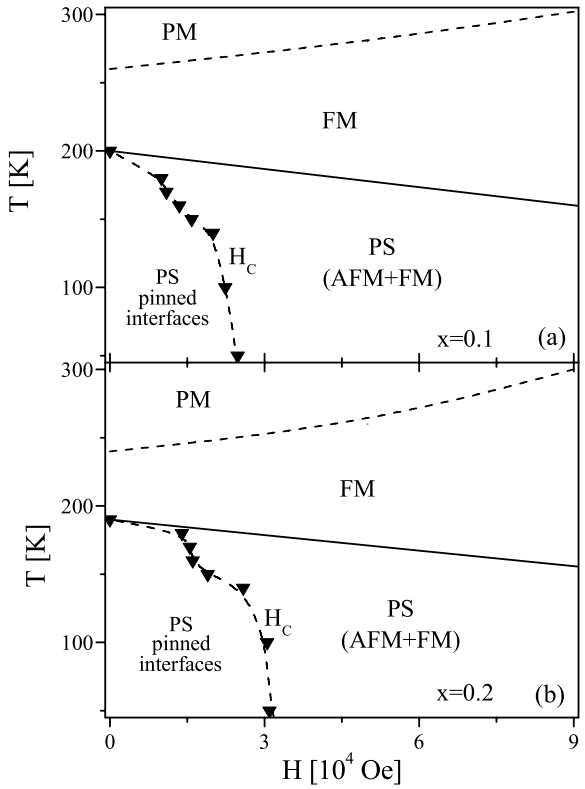

Figure 3. T–H phase diagram showing the different magnetic states of the samples, obtained from magnetic and transport measurements (taken from [21]), (a) $x = 0.1$ and (b) $x = 0.2$. Within the low temperature PS state, we included with solid triangles the critical field H_C, interpreted as the depinning field for the domain walls separating the FM and AFM clusters.

figure 4 we show that χ is very well described by a thermally activated behavior,

$$\chi(T) = \chi_0 + \chi_1 \, e^{-U/kT}, \tag{1}$$

where U is the activation energy that the interfaces must overcome in order to be released from the pinning potential, k is the Boltzmann constant, and χ_0 accounts for the non-activated contributions to the magnetic susceptibility. The lines shown along with the data points in figure 4 correspond to fits to this exponential law, from which we obtained $U \approx 104$ meV and 93 meV for $x = 0.1$ and 0.2, respectively.

Within the framework proposed here, where the linear behavior of $M(H)$ for $H < H_C$ is mainly dictated by the swelling of the FM clusters, we can also estimate the size increase of these FM domains. Since the spontaneous magnetization (M_0) is proportional to the FM volume [14, 21], we can write $\delta M_C/M_0 = 3\delta D_C/D$, where δD_C is the increase of the size of the FM clusters (of size D) when the applied field reaches H_C, and $\delta M_C = \chi H_C$. Combining these equations we can finally arrive at the relation

$$\chi = \left(\frac{3M_0}{H_C}\right) \times \frac{\delta D_C}{D}. \tag{2}$$

The most important consequence of this equation is that it provides a direct relation between three experimentally accessible parameters, namely χ, M_0, and H_C. Following equation (2), we constructed the plots of χ as a function of

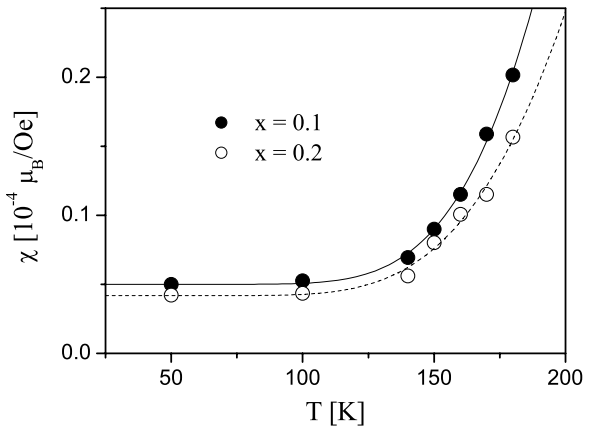

Figure 4. Temperature dependence of the magnetic susceptibility ($\chi = \partial M/\partial H$) obtained from the linear region of the $M(H)$ data of figure 1. The lines correspond to fits to the thermally activated law of equation (1).

the parameter $3M_0/H_C$ for both samples, as shown in figure 5 (each data point corresponds to a different temperature in the PS region). These data show a quite fair linear relation, with slopes $\delta D_C/D = 0.047$ and 0.038 for $x = 0.1$ and 0.2, respectively (a small $\chi \neq 0$ at $3M_0/H_C \to 0$ corresponds to the susceptibility of the AFM matrix). The obtained values indicate that, before the DWs are finally depinned at $H \sim$

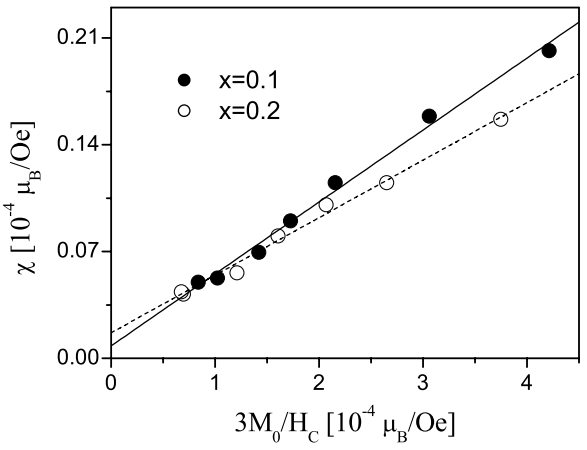

Figure 5. Susceptibility χ as a function of the scaling parameter $3M_0/H_C$. The lines show quite fair linear fits, as expected for the swelling of the FM clusters under an applied magnetic field.

H_C, through the thermally activated motion demonstrated in figure 4 the characteristic size of the FM clusters increases ∼4% in average.

Even though from magnetization data it is difficult to find the origin of the pinning mechanism, we can argue that point defects such as those producing quenched random disorder could hardly anchor extended elastic objects such as the magnetic interfaces. Indeed, collective pinning theories developed in the framework of vortices in HT_C superconductors predict that this kind of pinning would be extremely weak [36]. For this reason, although *a priori* it cannot be completely ruled out, pinning related to the A-site mismatch is very unlikely. Moreover, in the $Pr_{1-y}Ca_yMnO_3$ compound, since Pr^{3+} and Ca^{2+} have similar ionic radii, the A-site mismatch is almost completely eliminated. However, the sharp metamagnetic transition related to the burst increase of the FM domains is still observed in $M(H)$ at low temperatures [31]. Instead, the martensitic-like characteristics of this transition strongly suggest that strain due to lattice distortions is responsible for the pinning of the DWs. Indeed, neutron diffraction experiments in the sample $x = 0.2$ demonstrated that the FM and CO-AFM phases, even though they have the same *Pbnm* orthorhombic symmetry, present different structural characteristics (lattice parameters and atomic coordinates) [14]. This kind of crystallographic PS has also been reported in other manganese oxides [11, 15, 16], and certainly leads to lattice mismatch and distortions at the position of the magnetic DWs. Since these magnetic interfaces must also drag their structural deformations, the magnetization dynamics is necessarily affected. This would be comparable to the dynamics of lattice polarons, where the mobility of the charge carrier is reduced by the enlarged effective mass associated with the local distortions [5, 37]. In the case of the FM clusters, strain fields run all along the magnetic and structural interfaces and can be certainly much more effective than point defects to pin the DWs. Depinning of DWs must therefore proceed through the creation of surface excitations, i.e. thermal activation must create a bump in the magnetic interfaces, which can expand under the pressure of the magnetic field. This would be similar to the pinning of vortices by correlated defects in HT_C superconductors, with the difference of the dimensionality of the pinned entities. Within the usual pinning theory of elastic objects, the energy cost of generating such surface excitations is given by the so-called Larkin Hamiltonian [38, 39],

$$\mathcal{H} \approx \iint \left[\tfrac{1}{2}\varepsilon_t |\nabla u(\mathbf{r})|^2 + \varepsilon_p(\mathbf{r})\right] d^2r \tag{3}$$

where ε_t is the surface tension of the magnetic interface, ε_p the pinning energy per unit area, and u the amplitude of the excitation, which depends on the position $\mathbf{r}$ on the interface. For a surface excitation of amplitude ξ, simple integration gives, within factors of order unity, the approximate expression

$$\mathcal{H} \sim \varepsilon_t \xi^2 + \varepsilon_p L^2, \tag{4}$$

where L^2 indicates the area of the excitation on the magnetic and structural interface. However, L and ξ are not independent parameters. According to the well known Larkin's theory, the geometry of the excitation is determined by the competition between the elastic and pinning energies, such that the two terms in equation (4) are comparable [38, 39], giving the relation

$$\frac{\xi}{L} \sim \sqrt{\frac{\varepsilon_p}{\varepsilon_t}}, \tag{5}$$

as also happens in the pinning of vortices in HT_C superconductors [36]. Therefore, the excitation energy can be written as $\mathcal{H} \sim 2\varepsilon_t\xi^2$. For a complete description, the work done by the magnetic pressure must be added to the excitation energy. However, in this case we have to consider two types of excitation, either toward the inside of the FM cluster (a pit) or toward the outside (a bump) [40]. The Larkin Hamiltonian alone cannot distinguish between these two cases, but considering the magnetic pressure the total energy takes the form

$$\mathcal{H}_{p/b} \sim 2\,\varepsilon_t\,\xi^2 \pm mHL^2\xi, \tag{6}$$

where m is the magnetization per unit volume of the FM phase and the different signs describe the two excitations (+ for pits, $\mathcal{H}_p$, and − for bumps, $\mathcal{H}_b$). For $H = 0$, both excitations have equal probability, thus the average displacement of the domain walls is $\langle s\rangle = 0$. However, when a magnetic field is applied, bump excitations are favored and a net displacement of the DWs occurs, described by

$$\langle s\rangle \sim \xi(e^{-\mathcal{H}_b/kT} - e^{-\mathcal{H}_p/kT}), \tag{7}$$

k being the Boltzmann constant. In the linear regime ($H < H_C$), equation (7) can be written as $\langle s\rangle \sim e^{-\mathcal{H}/kT}H$, and because the volume of the cluster changes as $\delta V \propto D^2\langle s\rangle$, then the susceptibility depends as $\chi \sim e^{-\mathcal{H}/kT}$. Therefore, the excitation energy given by the Larkin model should be compared to the activation energy (U) obtained from the susceptibility data at low fields (figure 4).

Even though the DWs correspond to the interfaces between FM and AFM domains, the critical temperatures

of the two phases are not very different (they only differ by ∼20%, see figure 3), thus the exchange interactions and anisotropy energies are expected to be of the same order. Therefore, this gives the possibility to estimate the surface tension as the energy of a typical domain wall [40],

$$\varepsilon_t \sim 4\sqrt{\frac{JS^2K}{a}}, \tag{8}$$

where J is the exchange interaction between nearest neighbor spins of magnitude S, K is the magnetocrystalline anisotropy energy per unit volume, and a the distance between nearest neighbor spins. Appropriate values of these parameters are $a \approx 0.384$ nm (the Mn–Mn distance, see [28]), $JS^2 \sim 10^{-21}$ J (for critical temperatures ∼200–250 K), and $K \sim 10^4$–10^5 J m^{-3} (as observed in several doped manganites, see [41]). Thus, an estimation of the surface tension is $\varepsilon_t \sim 1.5$ mJ m^{-2}. Using the value of activation energy obtained from the fits in figure 4, $U \sim 100$ meV, the amplitude of the surface excitations can be estimated as $\xi \sim \sqrt{U/2\varepsilon_t}$, which gives $\xi \sim 2.3$ nm $\sim 6a$. In order to escape from the pinning site, the amplitude of the excitation must be the same as the range of the pinning potential. Therefore, if the interfacial structural distortions are responsible for the observed pinning effects, the strain fields associated with these magnetic and structural interfaces must also relax within ∼6 unit cells.

On the other hand, for the scaling proposed in equation (2) a linear relationship would be expected only if the parameter δD_C corresponds to some typical length scale of the system. When the magnetic field reaches the critical value H_C, the magnetic pressure is large enough so that the whole surface of the FM clusters is expected to reach the depinning distance, i.e. $\langle s \rangle \sim \xi$. In this situation, δD_C indicates the size of the excitations that allow the depinning of the interfaces, i.e. $\delta D_C \approx 2\xi \sim 4.6$ nm. Since $\delta D_C/D \sim 0.04$, we can estimate the size of the FM clusters as $D \sim 110$ nm. This sub-micrometer size is in very good agreement with direct observations obtained by scanning tunneling spectroscopy [7] and electron microscopy [8, 11] in other heavily doped manganites, and widely differs from the value $D \sim 1$ nm found in lightly doped manganites such as $La_{0.95}Ca_{0.05}MnO_3$ and $Ca_{0.98}La_{0.02}MnO_3$ [18], where electron-driven phase separation likely occurs.

Finally, according to Larkin's model, the critical field H_C corresponds to the driving force that overcomes the pinning and elastic forces [38, 39], such that $\mathcal{H}_b \sim 0$, i.e.

$$mH_CL^2 \sim \frac{U}{\xi}. \tag{9}$$

Using equation (5), we obtain the relation $\varepsilon_p \sim (mH_C\xi^3/U)\,\varepsilon_t$ that can be used to estimate the pinning energy. For our half-doped manganite $m = 3.5\ \mu_B/a^3$ and $H_C \approx 30$ kOe at low temperatures, thus $\varepsilon_p \sim 1.9$ mJ m$^{-2} \sim 1.3\,\varepsilon_t$. Therefore, the pinning energy is comparable to the surface tension of the magnetic interfaces, indicating that it is favorable for the system to create a magnetic DW in an already disordered and structurally distorted interface. Thus, as pointed out by different authors the structural strain appearing at the magnetic interfaces could be responsible for the occurrence of phase separation in heavily doped manganites [11, 30–32]. However, it should be noted that when strain effects are involved, the presence of grain boundaries or crystalline stress can change the physical behavior of the systems. In this sense, even though in our samples the grain size (∼3–4 μm) is much larger than the estimated size of the FM domains, quantitative comparisons with single crystals or epitaxial films may not be applicable.

4. Conclusions

The application of a magnetic field in phase-separated manganites produces an important decrease of the resistivity, the so-called colossal magnetoresistance effect. This behavior is related to the size increase of the FM clusters, favored by the Zeeman energy. However, we have shown that for $Pr_{0.5}Sr_{0.5-x}Ca_xMnO_3$ the pinning of the domain walls separating the FM and AFM phases gives rise to the appearance of two distinct regimes, whose boundary is the critical field H_C. When the magnetic field reaches this critical value the magnetic pressure overcomes the pinning force, thus releasing the DWs from the pinning centers and producing a rapid increase of the FM volume. For $H < H_C$ the $M(H)$ curves exhibit a linear dependence, with a thermally activated susceptibility whose activation energy $U \sim 100$ meV can be consistently described in terms of the elastic Hamiltonian given by Larkin's model. From the susceptibility data of this compound we also estimate that the FM domains increase by ∼4% before depinning occurs. Most likely, pinning of magnetic interfaces arises from the self-trapping due to the martensitic accommodation strain imposed by the structural mismatch between the FM and CO-AFM phases. Assuming this strain-related pinning mechanism we estimate a FM domain size of ∼110 nm, which involves ∼10^7 Mn sites, in agreement with previous observations. This type of interface strain could also be the origin of the phase separation phenomena.

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